National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2977

Mussel Tissue (Organic Contaminants and Trace Elements)

Standard Reference Material (SRM) 2977 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, methylmercury, and inorganic constituents in marine bivalve mollusk tissue and similar matrices. All of the constituents for which certified, reference, and information values are provided are naturally present in the freeze-dried mussel tissue. A unit of SRM 2977 consists of one bottle containing approximately 10 g of freeze-dried mussel tissue.

The development of this material was in response to the recommendations of the Group of Experts on Standards and Reference Materials (GESREM) established by the Intergovernmental Oceanographic Commission (IOC), United Nations Environment Program (UNEP), and the International Atomic Energy Agency (IAEA) [1]. The collection, preparation, and value assignment of SRM 2977 was a collaboration between the National Institute of Standards and Technology (NIST) and National Research Council of Canada (NRCC). Two additional mussel tissue materials have been developed as part of the GESREM recommendations: SRM 2976 Mussel Tissue (Trace Elements and Methylmercury) [2], which is intended for use in the determination of trace elements and methylmercury, and SRM 2978 Mussel Tissue (Organic Contaminants Raritan Bay, NJ) [3], which is intended for use in the determination of organic contaminants. These freeze-dried mussel tissue materials complement SRM 1974a Organics in Mussel Tissue (*Mytilus edulis*) [4], which is provided as a frozen tissue homogenate. SRM 2977 has concentrations of organic contaminants that are typically a factor of 2 to 4 lower than SRM 1974a and SRM 2978; the contaminant trace element concentrations in SRM 2977 are typically higher by a factor of 2 to 10 than the concentrations in SRM 2976.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 14 PAHs, 25 PCB congeners (some in combination), 7 chlorinated pesticides, 6 trace elements and methylmercury are provided in Tables 1 to 5. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more independent analytical techniques, and for selected compounds, results from a collaborating laboratory and from an interlaboratory comparison study. The certified values for the trace elements and methylmercury are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Concentration Values: Reference concentration values, expressed as mass fractions, are provided in Table 6 for 16 additional PAHs (some in combination). Reference concentration values are provided in Table 7 for nine additional inorganic constituents. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 6 and 7.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 8 for 23 additional trace elements. An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert.

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Certificate Issue Date: 01 May 2000

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The coordination of the technical measurements leading to certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.G. Vangel and M.S. Levenson of the NIST Statistical Engineering Division.

The mussels were collected under the supervision of A. Wagener from the Pontificia Unversidade Catolica, Do Rio De Janeiro, Brazil. The mussel tissue was freeze-dried at the Natural Products Support Group at the Frederick Cancer Research and Development Center (Frederick, MD) under the direction of T. McCloud. Preparation of the freeze-dried material was performed by M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program.

Analytical measurements at NIST were performed by M.J. Lopez de Alda, B.J. Porter, M.M. Schantz, S. Tutschku, and L. Yu of the NIST Analytical Chemistry Division.

Analytical measurements for selected PCB congeners were also performed at the Institute for National Measurement Standards, NRCC (Ottawa, Canada) by G. Gardner and C. Frasier. Results for selected PAHs, PCB congeners, and chlorinated pesticides were also used from 18 laboratories that participated in an intercomparison exercise coordinated by R. M. Parris of the NIST Analytical Chemistry Division. Analytical measurements for selected trace elements and methylmercury were also performed at the Institute of Applied Physical Chemistry, Research Centre Jülich (Jülich, Germany) by H. Emons and at the Department of Environmental Sciences, Jožef Stefan Institute (Ljubljana, Slovenia) by M. Horvat. Results for selected trace elements were also used from six laboratories that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, NRCC.

Expiration of Certification: The certification of this SRM lot is valid until **31 December 2009**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

NOTICE AND WARNING TO USERS

Storage: SRM 2977 is provided as a freeze-dried tissue homogenate in amber glass bottles. The tissue material should be stored at room temperature or below.

Handling: Normal biohazard safety precautions for the handling of biological tissues should be exercised.

Instructions for Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2977 are reported on a dry-mass basis. The freeze-dried mussel tissue homogenate is hygroscopic, and as received, contains greater than 3 % (mass fraction expressed as percent) residual moisture. The mussel tissue sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the mussel tissue should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The mussels (*Perna perna*, edible brown mussel) used for the preparation of SRM 2977 were collected in Guanabara Bay, Brazil. The mussels were shucked, and the tissue was shipped to NIST on dry ice in two batches, each containing approximately 35 kg. For processing, the tissue was allowed to partially thaw and was transferred into a Robot Coupe Vertical Cutter Mixer until it was half full. The mussel tissue was blended for 5 min into a puree form and then poured into metal trays and frozen. The material was then freeze-dried with a starting temperature of -10 °C and slowly warmed to a temperature of 10 °C. The dry material was broken into smaller chunks and then jet milled to produce a fine powder. The powder was blended for homogeneity by processing through the jet mill twice. The material was radiation sterilized (^{60}Co) and then aliquoted into jars (~ 10 g each).

[1]Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 2977 was similar to that reported for the recent certification of several environmental matrix SRMs [5-9] and consisted of combining results from analyses using various combinations of different extraction techniques, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM), clean up of the extracts using solid phase extraction (SPE), size exclusion chromatography (SEC), or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) for analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC) and (2) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on two stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl methylpolysiloxane phase and a 50 % (mole fraction) phenyl methylpolysiloxane phase.

Three sets of GC/MS results, designated as GC/MS (IA), GC/MS (IB), and GC/MS (II), were obtained using two columns with different selectivities for the separation of PAHs. For GC/MS (IA) analyses, duplicate subsamples of 3 g from eight bottles of SRM 2977 were extracted using PFE with DCM as described by Schantz et al. [10]. Size exclusion chromatography (SEC) on a preparative-scale divinylbenzene-polystyrene column (10 µm particle size, 10 nm (100 Å) pore size, 2.5 cm i.d. x 60 cm, PL-Gel, Polymer Labs, Inc., Amherst, MA) was used to remove the majority of the lipid and biogenic material. The extract was further fractionated using normal-phase LC [8-11] on a semi-preparative aminopropylsilane column to isolate the PAH fraction. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. x 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (IB) analyses were performed using the same processed extracts as for GC/MS (IA) with analysis by GC/MS using a 0.25 mm i.d. x 60 m fused silica capillary column with a 50 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-17, J&W Scientific, Folsom, CA). For the GC/MS (II) analyses, one sample (3 g) from each of three bottles was Soxhlet extracted for 18 h using DCM and a second sample (3 g) from the same three bottles was extracted using PFE with DCM. The SEC and normal-phase LC steps, as well as the GC/MS analysis, were the same as detailed for GC/MS (IA); however, the subsamples were extracted, processed, and analyzed as part of three different sample sets at different times using different calibrations for each set.

For the LC-FL analyses, subsamples of approximately 4 g from six bottles of SRM 2977 were Soxhlet extracted for 20 h using 200 mL of DCM. The extracts were concentrated and then processed through six aminopropylsilane SPE cartridges and a 0.2 µm inorganic membrane filter connected in series. The PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. x 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [11-14]. The isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. x 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [12,13]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the mussel tissue prior to solvent extraction for use as internal standards for quantification purposes. For the GC/MS (IA) and GC/MS (IB) measurements, selected ^{13}C-labeled PAHs were also added to the mussel tissue prior to solvent extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 2977 was used in 1997 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [15]. Results from 18 laboratories that participated in this exercise were used as the fifth data set in the determination of the assigned values for PAHs in SRM 2977. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PAHs.

Homogeneity Assessment for PAHs: The homogeneity of SRM 2977 was assessed by analyzing duplicate 3 g samples from eight bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (IA). No statistically significant differences among bottles were observed for the PAHs at the 3 g sample size.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 2977 was similar to that reported for the recent certification of several environmental matrix SRMs [6,8,16,17] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using SPE, SEC, or LC, followed by analysis using gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity and GC/MS on a third column.

Four sets of GC-ECD results were obtained and designated as GC-ECD (IA), GC-ECD (IB), GC-ECD (IIA), and GC-ECD (IIB). For the GC-ECD (IA) and GC-ECD (IB) analyses, duplicate 3 g subsamples from eight bottles of SRM 2977 were extracted using PFE with DCM. SEC was used to remove the majority of the lipid and biogenic material. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs

and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.25 mm x 50 m fused silica capillary column with a 10 % (mole fraction) octadecyl (C-18) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5/C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C-18 phase are designated as GC-ECD (IB). For GC-ECD (IIA) and GC-ECD (IIB), one sample (3 g) from each of three bottles was Soxhlet extracted for 18 h using DCM and a second sample (3 g) from the same three bottles was extracted using PFE with DCM. The SEC and normal-phase LC steps, as well as the GC-ECD analyses, were the same as detailed for GC-ECD (IA) and GC-ECD (IB); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Two sets of results were obtained by GC/MS: one at NIST and one at NRCC. For the analyses performed at NIST [GC/MS (NIST)], 8 g to 10 g subsamples from four bottles were Soxhlet extracted for 20 h with a mixture of 50 % hexane/50 % acetone. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The SPE step was repeated an additional two times. The extract was then analyzed by GC/MS using a 0.25 mm x 60 m fused silica capillary column with a proprietary phase (0.25 µm film thickness) (DB-XLB, J&W Scientific, Folsom, CA). For both the GC-ECD analyses, two PCB congeners that are not significantly present in the mussel extract (PCB 103 and PCB 198) [19,20], and 4,4'-DDT-d_8 were added to the mussel tissue prior to extraction for use as internal standards for quantification purposes. For the GC/MS analyses, PCB 103, PCB 198, and ^{13}C-labeled 4,4'-DDT, 4,4'-DDE, γ-HCH, PCB 28, PCB 52, PCB 101, PCB 118, PCB 153, PCB 138, and PCB 180 were added to the mussel tissue prior to extraction for use as internal standards for quantification purposes.

For the GC/MS analyses performed at the NRCC [GC/MS (NRCC)], 4 g subsamples from three bottles were Soxhlet extracted for 8 h with a mixture of 50 % hexane/50 % acetone. The chlorinated compounds were isolated from the extract using 2 % water-deactivated Florisil and alumina. The concentrated eluant was then analyzed using GC/high resolution MS (GC/HRMS) with a 5 % phenyl methylpolysiloxane phase (0.25 mm x 60 m, 0.25 mm film thickness). A solution containing ^{13}C-labeled PCB 28, PCB 52, PCB 101, PCB 118, PCB 105, PCB 153, PCB 138, PCB 180, and PCB 209 was added to the mussel tissue prior to extraction and a solution containing PCB 185 and ^{13}C-labeled PCB 169 was added to the sample just prior to GC/MS analysis for quantification purposes.

In addition to the analyses performed at NIST and NRCC, SRM 2977 was used in 1997 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [15]. Results from laboratories that participated in this exercise (17 laboratories for the PCB congeners and 14 laboratories for the chlorinated pesticides) were used as the seventh data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 2977. The laboratories participating in this exercise performed the analytical procedures routinely used in their laboratories to measure these analytes.

Homogeneity Assessment for PCB Congeners and Chlorinated Pesticides: The homogeneity of SRM 2977 was assessed by analyzing duplicate 3 g samples from eight bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC-ECD (IA). No statistically significant differences among bottles were observed for the PCB congeners and chlorinated pesticides at the 3 g sample size.

Analytical Approach for Inorganic Constituents: Value assignment of the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 2977 at NIST, NRCC, Research Centre Jülich, Jožef Stefan Institute, and six selected laboratories that participated in an interlaboratory comparison exercise coordinated by the NRCC [18]. A similar approach was used to provide certified and reference concentration values for trace elements in two other mussel tissue materials [2,4].

For the certified concentration values listed in Table 4, results were combined from analyses at NIST using inductively coupled plasma mass spectrometry (ICP-MS), analyses at NRCC using isotope dilution (ID) ICP-MS and graphite furnace atomic absorption spectrometry (GFAAS), analyses at Research Centre Jülich using one to four techniques, analyses at Jožef Stefan Institute using one or two techniques, and the mean of the results from six laboratories that participated in the NRCC interlaboratory comparison exercise. For the reference values provided in Table 7, results were combined from NIST, NRCC, Jožef Stefan Institute, Research Centre Jülich, and the NRCC interlaboratory comparison exercise. The information values in Table 8 are based on results of analyses at NRCC, Jožef Stefan Institute, and/or Research Centre Jülich. The analytical techniques used for the analysis of SRM 2977 for inorganic constituents are summarized in Table 9.

NIST Trace Element Analyses: The elements cadmium, cobalt, nickel, lead, copper, manganese, and strontium were determined using ICP-MS, quantified by the method of standard addition. Five mL of concentrated HNO_3 was added to 0.5 g subsamples from each of five bottles of SRM 2977. These samples were digested in closed vessels using programmed heating in

a microwave oven. The resulting tissue digests were quantitatively diluted into two concentration ranges; rhodium was added to each as an internal standard. The elements copper, manganese, and strontium were determined in the more dilute solution; cadmium, copper, nickel, and lead were determined in the more concentrated solution. Two spike solutions containing each of these group of elements were prepared and added to a split portion of each digest solution for the purpose of quantification by the method of standard addition. Prior to the quantitative determination of the analyte elements, an ICP-MS semi-quantitative analysis was performed to assess possible isobaric interferences. A correction was made for a molybdenum oxide interference on cadmium, the only interference observed.

NRCC Trace Element Analyses: Subsamples (0.25 g) from each of six bottles were placed in polytetrafluoroethylene (PTFE) digestion vessels with nitric acid; the vessels were sealed and heated in a microwave oven. (For the samples intended for ICP-MS analyses, a suitable amount of each enriched isotope solution was added to each sample prior to digestion). The digestion vessels were opened, (H_2O_2 was added to the samples for GFAAS and H_2O_2 and HF were added to the samples for ICP-Atomic Emission Spectrometry (AES), and the contents were evaporated to dryness. The residues were dissolved in nitric acid and double distilled water. The samples were analyzed by ID-ICP-MS for the determination of silver, cadmium, copper, nickel, lead, tin, and zinc. GFAAS was used for determination of silver, arsenic, cadmium, chromium, copper, nickel, and selenium, and ICP-AES was used for the determination of aluminum, iron, and zinc.

Research Centre Jülich Trace Element Analyses: The elements manganese, strontium, iron, zinc, calcium, magnesium, phosphorus, potassium, sodium, sulfur, and barium were determined by ICP-AES after pressure digestion (0.2 g of sample + 2 mL of HNO_3) in PTFE vessels. Aqueous acid-matched standard solutions containing scandium as an internal standard were used for calibration. The elements phosphorus and sulfur were determined without an internal standard. The elements cadmium, lead, and copper were determined in aliquots of corresponding digestion solutions by GFAAS using the method of standard addition [19,20]. ID-TIMS was used for the determination of cadmium, lead, copper, zinc, and thallium in solutions from pressure digestion (0.2 g of sample + 2 mL of HNO_3 + 0.2 mL HF) [21].

For mercury determination by cold vapor atomic absorption spectrometry (CVAAS), a subsample of 0.3 g to 0.5 g of material was digested with 10 mL concentrated nitric acid in heated quartz vessels closed with a cap [22]. The measuring system was calibrated using mercury (II) standard solutions in nitric acid. After high-pressure digestion (HPA) in quartz vessels (0.2 g of sample + 2 mL of HNO_3), cadmium, lead, copper, nickel, and thallium were determined by ICP-MS using aqueous standard solutions for calibration. In aliquots of HPA digestion solutions, electrochemical techniques were used for the determination of lead (differential pulse anodic stripping voltammetry (DPASV)), nickel (adsorptive stripping voltammetry (ADSV)), and selenium (cathodic stripping voltammetry (CSV)) at the hanging mercury drop electrode by standard addition method [23]. Selenium was quantified in HPA digestion and arsenic after open wet digestion (0.2 g of sample + 3 mL of HNO_3) by HG-AAS using aqueous standard solutions for calibration.

Jožef Stefan Institute Trace Element Analyses: Subsamples from each of six bottles of SRM 2977 were analyzed by electrothermal atomic absorption spectrometry (ETAAS), flame atomic absorption spectrometry (FAAS), instrumental neutron activation analysis (INAA), and radiochemical neutron activation analysis (RNAA). For the determination of trace elements by FAAS (iron, manganese, zinc, and copper) and ETAAS (cadmium, lead, and vanadium), subsamples of 300 mg were placed in PTFE Parr bombs with nitric acid and heated at 105 °C for 12 h. After digestion, the samples were equilibrated to room temperature and diluted with double distilled water.

For INAA, subsamples of 150 mg to 200 mg were sealed in plastic containers and irradiated for 20 h at a fluence rate of 1.0 x 10^{12} $cm^{-2} \cdot s^{-1}$. For the short-lived radionuclides, samples were irradiated for 1 min. The irradiated samples were transferred to clean polyethylene containers and counted after 2, 8, and 30 days. For the short-lived radionuclides, samples were counted at 2 min after irradiation for 5 min and at 3 h for 30 min. Samples were counted with a germanium detector. For the determination of mercury and selenium by RNAA, subsamples were sealed in quartz ampoules and irradiated for 16 h to 20 h at the fluence rate above. The samples were pyrolyzed resulting in volatilization of the mercury and selenium; selenium was trapped on soda lime and mercury was trapped on selenium-impregnated paper. The gamma activity of the isolated radionuclides was counted with a NaI(Tl) detector.

Methylmercury: The certified value for methylmercury is based on results of analyses of SRM 2977 at NIST and two other laboratories: Institute of Applied Physical Chemistry, Research Centre Jülich, (Jülich, Germany) and the Jožef Stefan Institute (Ljubljana, Slovenia). For the determination of methylmercury, SRM 2977 was analyzed at NIST using microwave digestion under acidic conditions, derivatization (phenylation), and preconcentration using solid phase micro-extraction (SPME) followed by GC with atomic emission detection (GC-AED). The GC-AED analyses were performed using a nonpolar 0.32 mm x 25 m fused silica capillary column with a polydimethylsiloxane phase (0.17 μm film thickness) (HP-1, Hewlett Packard, Wilmington, DE). For detection, the lines of mercury at 254 nm and carbon at 264 nm were used. A total of 14 subsamples (0.5 g to 1 g) from 6 bottles of SRM 2977 were analyzed at NIST. At the Research Centre Jülich, the analytical procedure for methylmercury

consisted of water steam distillation under acid conditions, anion exchange chromatographic separation of inorganic mercury and methylmercury, followed by CVAAS detection before and after ultraviolet radiation [24-26]. Triplicate subsamples (~300 mg) from each of three bottles were analyzed. Three methods were used for the determination of methylmercury at the Jožef Stefan Institute: (1) HCl extraction for 12 h anion exchange chromatographic separation of inorganic mercury and organomercury followed by cold vapor atomic absorption spectrometric detection before and after ultraviolet radiation (IEC-CVAAS) [24, 25, 27]; (2) H_2SO_4 extraction followed by ethylation at room temperature precollection, GC-pyrolysis with cold vapor atomic fluorescence spectrometric detection (GC-CVAFS) [28-31]; and (3) solid-liquid extraction into toluene followed by GC-ECD [27,30,32]. Six subsamples (200 mg to 500 mg) from one bottle of SRM 2977 were analyzed for each of the three analytical techniques and a subsample (500 mg) from each of six bottles of SRM 2977 were analyzed by one technique (GC-ECD).

Table 1. Certified Concentrations for Selected PAHs in SRM 2977

	Mass Fraction µg/kg (dry mass basis)[a]		
Fluorene[b,c,d,e,f]	10.24	±	0.43
Phenanthrene[b,c,d,e,f]	35.1	±	3.8
Fluoranthene[b,c,d,e,f]	38.7	±	1.0
Pyrene[b,c,d,e,f]	78.9	±	3.5
Benz[*a*]anthracene[b,c,d,e,f]	20.34	±	0.78
Benzo[*b*]fluoranthene[b,d]	11.01	±	0.28
Benzo[*e*]pyrene[c,d,e,f]	13.1	±	1.1
Benzo[*a*]pyrene[b,c,d,e,f]	8.35	±	0.72
Perylene[b,c,d,e,f]	3.50	±	0.76
Benzo[*ghi*]perylene[b,c,d,e,f]	9.53	±	0.43
Indeno[1,2,3-*cd*]pyrene[b,c,d,e,f]	4.84	±	0.81
Dibenz[*a,h*]anthracene[b,d]	1.41	±	0.19
Benzo[*b*]chrysene[b,c,d,e]	1.07	±	0.15
Picene[b,c,d,e]	2.29	±	0.27

[a] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [33]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [34], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM.

[c] GC/MS (IA) on 5 % phenyl methylpolysiloxane phase after PFE with DCM.

[d] GC/MS (IB) on 50 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/MS (IA).

[e] GC/MS (II) on 5 % phenyl methylpolysiloxane phase after PFE (3 samples) and Soxhlet (3 samples) with DCM.

[f] Results from 18 laboratories participating in an interlaboratory comparison exercise.

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 2977

			Mass Fraction µg/kg (dry mass basis)[b]		
PCB	8	(2,4'-Dichlorobiphenyl)[c,d,e,f,g,h]	2.10	±	0.15
PCB	18	(2,2',5-Trichlorobiphenyl)[c,d,e,f,g,h]	2.65	±	0.30
PCB	28	(2,4,4'-Trichlorobiphenyl)[c,d,e,f,g,h]	5.37	±	0.44
PCB	31	(2,4',5-Trichlorobiphenyl)[c,d,e,f]	3.92	±	0.24
PCB	44	(2,2'3,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h,i]	3.25	±	0.63
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h,i]	8.37	±	0.54
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)[d,f,g,h,i]	3.64	±	0.32
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)[c,d,e,f,i]	2.15	±	0.10
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)[d,f,g,h,i]	5.39	±	0.59
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl) [c,d,e,f,i]	1.59	±	0.20
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)[c,d,e,f,g,h,i]	11.2	±	1.2
	90	(2,2',3,4',5-Pentachlorobiphenyl)			
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)[c,d,e,f,g,h,i]	3.76	±	0.49
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl) [c,d,e,f,i]	4.03	±	0.20
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)[c,d,e,f,g,h,i]	10.5	±	1.0
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)[c,d,e,f,g,h,i]	2.49	±	0.28
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)[c,d,e,f,g,h]	16.6	±	1.6
	163	(2,3,3',4'5,6-Hexachlorobiphenyl)			
	164	(2,3,3',4',5',6-Hexachlorobiphenyl)			
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl)[c,d,e,f,i]	9.23	±	0.12
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)[c,d,e,f,i]	3.07	±	0.18
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)[c,d,e,f,g,h,i]	14.1	±	1.0
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)[c,d,e,f,i]	0.960	±	0.085
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)[c,d,e,f,g,h,i]	2.95	±	0.23
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)[c,d,e,f,g,h,i]	6.79	±	0.67
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)[c,d,e,f,i]	1.33	±	0.10
PCB	187	(2,2',3,4',5,5'6-Heptachlorobiphenyl)[c,d,e,f,g,h,i]	4.76	±	0.38
	159	(2,3,3',4,5,5'-Hexachlorobiphenyl)			
	182	(2,2,'3',4,4',5,6'-Heptachlorobiphenyl)			
PCB	194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)[c,d,e,f]	0.897	±	0.042

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [35] and later revised by Schulte and Malisch [36] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the GC analysis conditions used, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [33]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [34], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[c] GC-ECD (IA) on 5 % phenyl methylpolysiloxane phase after PFE with DCM.

[d] GC-ECD (IB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IA).

[e] GC-ECD (IIA) on 5 % phenyl methylpolysiloxane phase after PFE (3 samples) and Soxhlet extraction (3 samples) with DCM.

[f] GC-ECD (IIB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

[g] NRCC data from GC/HRMS on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[h] Results from 17 laboratories participating in an interlaboratory comparison exercise.

[i] GC/MS on a proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 2977

	Mass Fraction[a] µg/kg (dry mass basis)
cis-Chlordane (α-Chlordane)[b,c,d,e,f,g]	1.42 ± 0.13
trans-Nonachlor[b,c,d,e,f,g]	1.43 ± 0.10
Dieldrin[b,c,e,f,g]	6.04 ± 0.52
4,4'-DDE[b,c,d,e,f,g]	12.5 ± 1.6
2,4'-DDD[b,c,d,e,f,g]	3.32 ± 0.29
4,4'-DDD[b,c,d,e,f,g]	4.30 ± 0.38
4,4'-DDT [b,c,e,f,g]	1.28 ± 0.18

[a] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [33]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [34], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] GC-ECD (IA) on 5 % phenyl methylpolysiloxane phase after PFE with DCM.

[c] GC-ECD (IB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IA).

[d] GC/MS on a proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[e] GC-ECD (IIA) on 5 % phenyl methylpolysiloxane phase after PFE (3 samples) and Soxhlet extraction (3 samples) with DCM.

[f] GC-ECD (IIB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

[g] Results from 14 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Inorganic Constituents in SRM 2977

Element	Degrees of Freedom	Mass Fraction mg/kg (dry mass basis)[a]
Cadmium[b,c,d,e,f,g,h]	4	0.179 ± 0.003
Copper[b,c,d,e,f,g,h,j]	5	9.42 ± 0.52
Lead[b,c,d,e,f,g,h,k,l]	4	2.27 ± 0.13
Manganese[b,i,m]	2	23.93 ± 0.29
Nickel[b,c,d,e,g,n]	4	6.06 ± 0.24
Strontium[b,i,m]	2	69.3 ± 4.2

[a] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the mean of three to six results from the following: (1) the mean of ICP-MS analyses performed at NIST; (2) the mean of ID-ICP-MS analyses performed at NRCC; (3) the mean of GFAAS analyses performed at NRCC; (4) the mean of results from six selected laboratories participating in the NRCC intercomparison exercise; (5) the mean of results from analyses by HGAAS, GFAAS, ICP-MS, ID-TIMS, ADSV, and/or DPASV performed at Research Centre Jülich; and (6) the mean of results from analyses by INAA, GFAAS, and FAAS performed at Jožef Stefan Institute. The expanded uncertainty in the certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO Guide [34] and k is the coverage factor. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of all the uncertainties in the certified value. Here u_c is given by the standard error of the mean of the available values.

[b] Measured at NIST using ICP-MS.

[c] Measured at NRCC using ID-ICP-MS.

[d] Measured at NRCC using GFAAS.

[e] Measured by six laboratories as part of the NRCC interlaboratory comparison exercise.

[f] Measured at Research Centre Jülich using GFAAS

[g] Measured at Research Centre Jülich using ICP-MS.

[h] Measured at Research Centre Jülich using ID-TIMS.

[i] Measured at Jožef Stefan Institute using INAA.

[j] Measured at Jožef Stefan Institute using FAAS.

[k] Measured at Jožef Stefan Institute using GFAAS.

[l] Measured at Research Centre Jülich using DPASV.

[m] Measured at Research Centre Jülich using ICP-AES.

[n] Measured at Research Centre Jülich using ADSV.

Table 5. Certified Concentration of Methylmercury in SRM 2977

	Mass Fraction μg/kg (dry mass basis)
Methylmercury[a,b]	36.2 ± 1.7

[a] Results for methylmercury are reported as μg/kg mercury.

[b] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the mean of results from five different laboratory analyses of SRM 2977 using five different analytical methods. The expanded uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [34] and k is the coverage factor. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of all the uncertainties in the certified value. Here u_c is given by the standard error of the mean of the five analyses. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with four degrees of freedom.

Table 6. Reference Concentrations for Selected PAHs in SRM 2977

The values in Table 6 are provided as reference values because: either the results have not been confirmed by an independent analytical technique as required for certification, or the disagreement among the methods was greater than expected for certified values. These reference values should be useful for comparison with results obtained using similar procedures.

	Mass Fraction[a] μg/kg (dry mass basis)
Naphthalene[b,c,d,e]	19 ± 5
1-Methylnaphthalene[b,c,d,e]	16 ± 5
2-Methylnaphthalene[b,c,d,e]	18 ± 5
Biphenyl[b,c,d,e]	6.8 ± 0.6
Acenaphthene[b,c,d,e]	4.2 ± 0.4
Anthracene[b,c,d,e,f]	8 ± 4
1-Methylphenanthrene[b,c,d,e]	44 ± 2
2-Methylphenanthrene[b,c,d]	43 ± 1
3-Methylphenanthrene[b,c,d]	44.2 ± 0.4
4-Methylphenanthrene/9-Methylphenanthrene[b,c,d]	36 ± 2
Benzo[*c*]phenanthrene[b,c,d]	9.4 ± 0.3
Chrysene[f]	49 ± 2
Triphenylene[f]	39 ± 1
Benzo[*j*]fluoranthene[c]	4.6 ± 0.2
Benzo[*k*]fluoranthene[b,c,d,f]	4 ± 1
Dibenz[*a,h*]anthracene/Dibenz[*a,c*]anthracene[b,d,e]	2.0 ± 0.2

[a] The results are expressed as the reference value ± the expanded uncertainty. Each reference value is a mean of the means from one to four analytical methods, weighted as described by Paule and Mandel [33]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [34], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] GC/MS (IA) on 5 % phenyl methylpolysiloxane phase after PFE with DCM.

[c] GC/MS (IB) on 50 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/MS (IA).

[d] GC/MS (II) on 5 % phenyl methylpolyxiloxane phase after PFE (3 samples) and Soxhlet (3 samples) with DCM.

[e] Results from 18 laboratories participating in an interlaboratory comparison exercise.

[f] LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 2977 as Determined by Multiple Laboratories

The values in Table 7 are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, the agreement among results from multiple methods was insufficient for certification, or insufficient analyses have been performed at NIST to confirm the results of the external laboratories.

Element	Degrees of Freedom	Mass Fraction mg/kg (dry mass basis)[a]
Arsenic[b,c,d,e]	3	8.83 ± 0.91
Chromium[b,c,d]	2	3.91 ± 0.47
Cobalt[c,f,g]	2	0.48 ± 0.13
Iron[b,c,d,h]	4	274 ± 18
Mercury[d,i,j,k,l]	3	0.101 ± 0.004
Selenium[b,c,d,e,k,m]	3	1.78 ± 0.16
Silver[b,c,d,n]	3	4.58 ± 0.33
Tin[d,n]	5	1.47 ± 0.27
Zinc[b,c,d,h,n,p]	4	135 ± 5

[a] The results are expressed as the reference value ± the expanded uncertainty. The reference value is the mean of three to five values from the following values: (1) the mean of ID-ICP-MS analyses performed at NIST; (2) the mean of ID-ICP-MS analyses performed at NRCC; (3) the mean of GFAAS or ICP-AES analyses performed at NRCC; (4) the mean of results from five or six selected laboratories participating in the NRCC intercomparison exercise; (5) the mean of results from analyses by CSV, HGAAS, ICP-AES, and/or ID-TIMS performed at Research Centre Jülich; and (6) the mean of results from analyses by CVAAS, INAA, and/or RNAA performed at Ljubljana. The expanded uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [34] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c is given by the standard error of the mean of the available values. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with four degrees of freedom.

[b] Measured at NRCC using GFAAS or ICP-AES.

[c] Measured at Jožef Stefan Institute using INAA.

[d] Measured by five or six laboratories as part of the NRCC interlaboratory comparison exercise.

[e] Measured at Research Centre Jülich using HGAAS.

[f] Measured at NIST using ICP-MS.

[g] Measured at Research Centre Jülich using ICP-MS.

[h] Measured at Research Centre Jülich using ICP-AES.

[i] Measured at NRCC using CVAAS.

[j] Measured at Research Centre Jülich using CVAAS.

[k] Measured at Jožef Stefan Institute using RNAA.

[l] Measured at Jožef Stefan Institute using CVAAS.

[m] Measured at Research Centre Jülich using CSV.

[n] Measured at NRCC using ID-ICP-MS.

[o] The reference value for tin is the mean of the results from NRCC using ID-ICP-MS and the individual results from five laboratories participating in the NRCC interlaboratory exercise (n = 6).

[p] Measured at Research Centre Jülich using ID-TIMS.

Table 8. Information Values for the Concentrations for Selected Inorganic Constituents in SRM 2977

Element	Mass Fraction
	% (dry mass basis)
Calcium[a,b]	0.83
Chlorine[b]	4.3
Magnesium[a,b]	3.9
Phosphorus[a]	1.1
Potassium[a,b]	1.2
Sodium[a,b]	2.4
Sulfur[a,b]	2.9
	mg/kg (dry mass basis)
Aluminum[b,c]	400
Antimony[b]	0.048
Barium[a]	4.7
Bromine[b]	215
Cerium[b]	0.93
Cesium[b]	0.039
Gold[b]	0.013
Iodine[b]	26
Lanthanum[b]	0.44
Rubidium[b]	6.7
Samarium[b]	0.064
Scandium[b]	0.055
Thorium[b]	0.19
Uranium[b]	0.083
Vanadium[b]	1.1
	µg/kg (dry-mass basis)
Thallium[d,e]	10.2

[a] Measured at Research Centre Jülich using ICP-AES.
[b] Measured at Jožef Stefan Institute using INAA.
[c] Measured at NRCC using ICP-AES.
[d] Measured at Research Centre Jülich using ICP-MS.
[e] Measured at Research Centre Jülich using ID-TIMS.

Table 9. Analytical Methods Used for the Analysis of SRM 2977 for Inorganic Constituents

Elements	Analytical Methods
Aluminum	ICP-AES, INAA
Antimony	INAA
Arsenic	CSV, GFAAS, HGAAS, ICP-AES, ICP-MS, INAA
Barium	ICP-AES
Bromine	INAA
Cadmium	FAAS, GFAAS, ICP-MS, ICP-AES, ID-ICP-MS, ID-TIMS
Calcium	INAA, ICP-AES
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	GFAAS, ICP-MS, INAA
Cobalt	ICP-MS, INAA, RNAA
Copper	FAAS, GFAAS, ICP-AES, ICP-MS, ID-ICP-MS, ID-TIMS
Iodine	INAA
Iron	FAAS, ICP-AES, ICP-MS, INAA
Lanthanum	INAA
Lead	DPASV, GFAAS, ICP-MS, ID-TIMS, XRF
Magnesium	ICP-AES, INAA
Manganese	FAAS, ICP-AES, ICP-MS, INAA
Mercury	CVAAS, ICP-MS INAA, RNAA
Nickel	ADSV GFAAS, ICP-AES, ICP-MS
Potassium	ICP-AES, INAA
Rubidium	INAA
Samarium	INAA
Scandium	INAA
Selenium	CSV, GFAAS, HGAAS, ICP-MS, INAA, RNAA
Silver	GFAAS, ID-ICP-MS, ICP-MS, INAA
Sodium	ICP-AES, INAA
Strontium	ICP-MS, HG-AAS, INAA
Sulfur	ICP-AES, INAA
Thallium	ICP-MS, ID-TIMS, INAA
Tin	GFAAS, ICP-AES, ICP-MS, ID-ICP-MS
Thorium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICP-AES, ICP-MS, ID-ICP-MS, ID-TIMS, XRF, INAA

Methods

ADSV	Adsorptive stripping voltammetry
CSV	Cathodic stripping voltammetry
CVAAS	Cold vapor atomic absorption spectrometry
DPASV	Differential pulse anodic stripping voltammetry
ETAAS	electrothermal atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ID-ICP-MS	Isotope dilution inductively coupled plasma mass spectrometry
ID-TIMS	Isotope dilution thermal ionization mass spectrometry
INAA	Instrumental neutron activation analysis
RNAA	Radiochemical neutron activation analysis
XRF	X-ray fluorescence spectrometry

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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (30l) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the internet http://www.nist.gov/srm.

APPENDIX A

The laboratories listed below participated in the interlaboratory comparison exercise coordinated by NRCC for the determination of trace elements in SRM 2977.

Analytical Services Laboratories Ltd., Vancouver, British Columbia, Canada
Battelle Pacific Northwest, Sequim, WA, USA
Can Test Ltd., Vancouver, British Columbia, Canada
Florida Institute of Technology, Division of Marine and Environmental Systems, Melbourne, FL, USA
Massachusetts Water Resources Authority, Winthrop, MA, USA
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